METHOD AND APPARATUS FOR SIMULTANEOUS MEASUREMENT OF THE REFRACTIVE INDEX AND THICKNESS OF THIN FILMS

TECHNICAL FIELD

The present invention relates to a beam deflection technique for simultaneous measurements of the thickness, refractive index and optical absorption of transparent materials using a charge coupled device (CCD) camera. The method and apparatus is particularly suited to measuring flat, thin materials.

BACKGROUND OF THE INVENTION

Many optical devices such as spectrophotometers use absorption spectroscopy to measure the concentration of various materials. There are two primary procedures used for measuring concentration. An absorption value of a material is calculated at an absorbing wavelength and the absorption of the material at a minimally absorbing wavelength is subtracted. This process of "blanking" minimizes uncertainties due to sample cell imperfections and parasitic scattering. Another procedure involves taking absorption measurements on a material at various time intervals and analyzing the differences between measurements. In these methods, as well as in other technologies such as scattering techniques and optical cavity based techniques, the accurate knowledge of the refractive index of the material and optical path length is critical information.

In these methods of concentration measurement, the reflectivity losses due to the index of refraction are generally assumed to be a function only of wavelength. This assumption introduces uncertainties in absorption measurements because the refractive index also directly depends on the number density and the type of polarizable species in the material. As a result, there is a need to develop techniques to measure accurately refractive indices of samples at the specific optical absorption wavelength.

Accurate thickness (optical path length) data for a given material greatly enhances the sensitivity and accuracy of spectrophotometers and other absorption devices in concentration measurement. Furthermore, the need to accurately monitor and control the refractive index and thickness of samples *during production* exists in the manufacturing of materials such as laboratory windows, optical lenses, automotive parts, and optical glasses.

The refractive index and optical path are measured by a method described in U.S. Patent No. 6,057,928, issued to Li et al. This technique measures the variations in reflectance (ratio of the reflected beam over the incident beam powers) and the beam phase distribution as a function of the incidence angle of a far IR beam on a film. Using Fresnel equations relating the reflectance and the phase of a beam to the incidence angle and the refractive index, the refractive index of a film is estimated. The refractive index is determined by fitting the experimental reflectance and phase variation curves to the theoretical Fresnel equations.

The method in the '928 patent uses GHz-THz radiation sources in the far IR region. These sources have wavelengths of 0.1 mm to 1 cm, and therefore the technique is available for materials of a few microns in thickness. The method described in the '928 patent uses a femtosecond mode-locked laser to excite an emitter, and a sophisticated detection mechanism.

The need remains for a cost-effective technique that provides for the simultaneous measurement of the refractive index and thickness of various film materials.

SUMMARY OF THE INVENTION

The present invention provides a method for the simultaneous determination of sample thickness **L** and index of refraction **n**. The method comprises forming a sample with first and second surfaces. A radiation beam is also formed and the radiation beam

impinges onto the sample at an incidence angle (A_1) relative to the perpendicular axis to the first surface. The method also includes reflecting the impinged radiation beam from the first and second surfaces of the sample to form first and second reflected radiation beams. The first and second reflected radiation beams then impinge on a detection device which allows the measurement of the distance (d_1) on the detector between the impingement point of the first reflected beam and the impingement point of the second reflected radiation beam. The method further includes altering the first incidence angle to a second incidence angle (A_2) and measuring the distance (d_2) between the impingement point of a third reflected beam and the impingement point of a fourth reflected beam on the detection device. The method finally provides for obtaining the sample thickness **L** and sample index of refraction **n** from the following equations:

$$d_1 = [2.\mathbf{L}/\mathbf{n}].[\sin A_1/(1-(\sin^2 A_1)/\mathbf{n}^2)^{1/2}] \text{ and}$$

$$d_2 = [2.\mathbf{L}/\mathbf{n}].[\sin A_2/(1-(\sin^2 A_2)/\mathbf{n}^2)^{1/2}]$$

Another method for the simultaneous determination of a sample thickness **L** and index of refraction **n**, is also provided which involves transmitting the radiation beam through the sample, and intercepting the transmitted radiation beam by the detection device and measuring the distance (d_1) between the point on the detection device where the axis intercepts the detection device and the point on the detection device where the transmitted beam impinges on the detection device. This method also includes directing the radiation beam along a second axis with the sample, and transmitting the radiation beam through the sample and measuring a second distance (d_2) between a point on the detection device where the second axis intercepts the detection device and a point on the detection device where the transmitted beam impinges on the detection device. This method involves solving the following system of equations for transmitted radiation beams:

$$d_1 = \mathbf{L}\ [\ \sin A_1 - (\sin 2A_1 \div 2(\mathbf{n}^2-\sin^2 A_1)^{1/2})] \text{ and}$$

$$d_2 = \mathbf{L}\,[\,\sin A_2 - (\sin 2A_2 \div 2(\mathbf{n}^2 - \sin^2 A_2)^{1/2})]$$

to obtain values for **L** and **n**.

BRIEF DESCRIPTION OF THE DRAWINGS

The invention can be more fully understood from the following description thereof in connection with the accompanying drawings described as follows.

FIG. 1 is a schematic view of a light beam transmitted through a film.

FIG. 2 is a schematic view of a light beam reflected by a film.

FIG. 3 is a schematic view of a measurement apparatus of the present invention.

FIG. 4 is a schematic view of a measurement of a liquid sample in a cuvette according to the present invention.

DETAILED DESCRIPTION OF THE INVENTION

The method and apparatus of the present invention provide for the simultaneous measurement of a film thickness and index of refraction by using a photo detector and applying Snell's Law of refraction.

By using photoimaging, such as with a charge coupled device (CCD), the present invention allows measuring the refractive index (n) and the local thickness (L) of flat samples such as glass, sapphire, quartz or plastic windows. The materials to be measured are preferably transparent in the visible region of radiation. This technique is based on the deflection property of an oblique laser beam when crossing a material with a different refractive index than that of the medium in which it is located. Typically the measurement of the properties of materials is done in an atmosphere of air, which has a refractive index of one ($n_{air} = 1$). The material being measured typically has a refractive

index greater than one, and the change of refractive index at the air-material interface causes a change in the direction of propagation of the light beam.

A change in propagation direction, or deflection, occurs at two air-material interfaces of the sample studied, the interface as the light beam enters the material, and the interface when the light beam exits the material. If the laser beam forms an incident angle with the vector normal to the material surfaces, the amount of deflection is a function of the incident angle, the refractive index of the material, and the thickness of the material.

By using Snell's law of refraction at each air-material interface, $n_1 \sin\theta_1 = n_2 \sin\theta_2$, where the subscripts 1 and 2 stand for the incident media (air) and the sample material, respectively, an expression for the beam displacement can be derived. For the purposes of this derivation, the two air-material interface surfaces of the samples are considered to be at least locally parallel. If the interface surfaces are parallel than θ_1 and θ_2 are the same angle, referred to hereafter as α. The application of Snell's law to the film configuration provides the following expression:

$$\mathbf{d = L.\left[\sin\alpha - \frac{\sin 2\alpha}{2.\sqrt{N^2 - \sin^2\alpha}}\right]}$$

EQN. 1

Equation 1 relates the thickness of the sample material, **L**, and the refractive index of the material, **n**, to the incidence angle, α, and the deflected beam displacement, d. It's clear that if either **L** or **n** is known than the other parameter can be directly estimated through EQN 1. However, when the two parameters, **L** and **n**,

are both unknown, interferometric techniques are typically used to estimate the optical constants simultaneously.

However, if at least two measurements are performed at two different incident angles, α_1, and α_2, **L** and **n** can be simultaneously measured directly using a photodetector device that can measure d_1 and d_2, such as a CCD camera.

The invention will next be described with reference to the figures in which same numbers indicate same parts in all figures. The figures are provided to facilitate the description of the invention and are not exact representations to scale of the different elements depicted nor do they show additional elements that are not essential in describing the present invention.

The deflection phenomenon exploited by the present invention is diagramed in FIG. 1. A light beam source 12, such as a laser, provides an incident light beam 13 on a film sample 22. The two faces of the sample 20 and 24 are parallel, at least locally for the light beam, and therefore the beam is deflected by the film, and exits the film in a beam 15 parallel to the incident beam 13.

The incident beam 13 makes an angle 16, labeled herein as α, with a vector 18 normal to the film surface 20. Measurement of transmitted light from a light source polarized in the incidence plane should be achieved with incident angles corresponding to an internal angle (on the second interface) smaller than the total internal reflection angle. An incident angle corresponding to the Brewster angle is preferable in this case. With non-polarized beam sources, any incident angle provides a transmitted beam 15. The transmitted beam is deflected while propagating through the thickness of the film 14 because of the film has a different index of refraction. The transmitted beam is again deflected at the material-air interface at the second film surface 24. The transmitted beam 15 forms the same angle α with the vector normal to the second surface of the film 24. The deflection of the beam while propagating through the film is a function of the index of refraction of the film material and the thickness of the

material, as well as the incident angle. The deflection can be measured as the distance between the parallel trajectories 28 of the incident beam 13 and the transmitted beam 15. As shown in FIG. 1, a detector is positioned to intercept the non-deflected beam 13, as well as the deflected beam 15.

One method for the simultaneous determination of a sample thickness **L** and index of refraction **n**, provided herein is based in part on the principles illustrated in FIG. 1. Since both **n** and **L** are unknowns in EQN 1, two measurements of deflection 28, collected at two different incident angles α, provides two expressions that satisfy EQN 1 with two remaining unknowns, **n** and **L**. With at least two measurements, the expressions can be solved for accurate values of both **n** and **L**.

The method includes directing a radiation beam along an axis to form a first angle (A_1) with the sample 22. The transmitted radiation beam intercepts a detection device, which measures beam deflection as the distance (d_1) between the point of the radiation beam axis and the point of the transmitted beam.

A second measurement is collected by directing the radiation beam along a second axis forming a second angle (A_2) with the sample. Again the transmitted beam intercepts the detection device and a second distance (d_2) between the point the second axis and the point of the transmitted beam is measured. With the angle and deflection data collected (A_1, A_2, d_1, and d_2), the following system of equations, from EQN. 1 may be solved:

$$d_1 = \mathbf{L}\,[\,\sin(A_1) - (\sin(2A_1) \div 2(\mathbf{n}^2 - \sin^2(A_1))^{1/2})] \text{ and}$$

$$d_2 = \mathbf{L}\,[\,\sin(A_2) - (\sin(2A_2) \div 2(\mathbf{n}^2 - \sin^2(A_2))^{1/2})]$$

to obtain values for **L** and **n**.

A similar method can be used to obtain **L** and **n** values by measuring the deflection of radiation beams reflected off a film sample. Such a measurement arrangement is shown schematically in FIG. 2. A radiation source 12 transmits a beam 13 at an incident angle 16 to a vector 18 normal to the surface 20 of the film sample 22.

The beam is partially reflected at the first surface of the film sample 17, and the position of the reflected beam is measured at a detector 26. The beam 13 is also partially transmitted 17' through the width of the sample 14. The transmitted beam 17' is deflected during propagation through the sample due to the change of index of refraction as discussed above. The transmitted beam 17' encounters the second surface 24 of the sample, and a portion of the transmitted beam 19 is reflected off the second surface 24. The beam reflected off the second surface 19 encounters the first surface of the sample 20, and a portion of the beam is transmitted through the surface of the sample, encountering another change in the index of refraction. This transmitted beam 19' is parallel to the first reflected beam 17, and the position of the resulting beam 19' is measured at the detector 26. The amount of deflection is measured as a distance 28 between the two positions from the reflected beams 17 and 19'.

The angle of reflection is dictated by the angle of incidence, as the angle of reflection equals the angle of incidence, so that there is only a single angle contributing to the calculation of **L** and **n** using measurements collected through beam reflection.

A method for the simultaneous determination of a sample thickness **L** and index of refraction **n**, by measuring the position of reflected radiation beams includes transmitting a radiation beam onto the sample at a first incidence angle (A_1) relative to an axis 18 perpendicular to the first surface 20 of the sample. The beam is reflected from both the first 20 and second 24 surfaces of the sample and generates first 17 and second 19' reflected radiation beams. A detection device 26 measures the relative positions of the first and second reflected radiation beams. From the relative positions, a distance (d_1) on the detector can be measured. A second measurement is taken by altering the first incidence angle (A_1) to a second incidence angle (A_2) and again

measuring the distance (d_2) between a third reflected beam and a fourth reflected beam on the detection device. Values for the sample thickness **L** and sample index of refraction **n** can then be calculated using the following expressions, derived from Snell's law for reflected beams:

$$d_1 = [2\mathbf{L}/\mathbf{n}][\sin(A_1) \div (1 - (\sin^2(A_1))/\mathbf{n}^2)^{1/2}] \text{ and}$$

$$d_2 = [2\mathbf{L}/\mathbf{n}][\sin(A_2) \div (1 - (\sin^2(A_2))/\mathbf{n}^2)^{1/2}].$$

As with the transmitted beam calculation, measurements obtained at two incident angles provide the above two equations with two unknowns, which can be solved directly.

An exemplary apparatus for collecting these measurements is shown schematically in FIG. 3. A radiation source 12 provides a beam which may be polarized using polarization optics 30, and directed towards a sample 22 using a series of optical mirrors 32 and 32'. The sample 22 is secured onto a sample stage 34, that can rotate about an axis to vary the angle 16 between the vector 18 normal to the sample surface. The sample stage also allows for easy transfer of samples, such as on a production line. When measurements are performed in the transmission mode, as shown in FIG. 3, the non-deflected beam position may be gathered when the sample stage does not contain a sample, either before or after samples are loaded onto the stage. Alternate configurations may include a sample with a fixed position with optics, or multiple sources providing radiation beams at various angles to the sample surface. The radiation sources and the detectors may also move in space to optimize the collection of beam deflection data.

The exemplary apparatus in FIG. 3 shows a detector 26 positioned to intercept the non-deflected and deflected transmitted beams, and connected to a computer with a display 38 to show the positions of the deflected and non-deflected beams 35 and 36, and the measured distance between the beams 28.

A single beam with a non-rotating sample may also be used by configuring the incident beam with different sagittal and azimuthal angle values α and θ. The corresponding displacements, d_α and d_θ are then measured horizontally and vertically.

In practice a method using sagittal and azimuthal angle values may express the angle values in a Cartesian coordinate system for convenience. In such a case, a substantially monochromatic collimated radiation beam is transmitted onto the sample along an axis forming a first angle A_x and subsequently a second angle A_y in a coordinate system having the sample in a plane defined by x and y axes. In this system, the angle A_x is measured in a plane defined by the x and z axes and an angle A_y is measured in a plane defined by the y and z axes. The detector can be located in a plane parallel to the x-y plane and the first distance d_x is measured on the x-axis. A second distance d_y can be measured on the y-axis and the measurements used to solve the following equations, as stated above:

$$d_x = \mathbf{L} [\sin(A_x) - (\sin(2A_x) \div 2(\mathbf{n}^2 - \sin^2(A_x))^{1/2})] \text{ and}$$

$$d_y = \mathbf{L} [\sin(A_y) - (\sin(2A_y) \div 2(\mathbf{n}^2 - \sin^2(A_y))^{1/2})]$$

to obtain values for **L** and **n**. Similar conversion may be performed for the measurements collected in the reflection mode.

The radiation source may be a laser, light emitting diode (LED) or an incoherent source, among others. As such the radiation beam may be monochromatic, and or collimated, however sources need not be either monochromatic or collimated. Low-coherence and incoherent sources avoid possible interference between the reflected beams, which may influence the exact positioning of the beams' spots on the CCD.

As mentioned above, for measurements in the transmission mode, the incident angle α must lead to an internal angle on the second interface smaller than the total internal reflection angle (Critical angle) when polarized radiation sources are used. For

these measurements the radiation beam is preferably at an incident angle α greater than 10 degrees to the sample surface for resolution purposes.

The sample for analysis is described as having a first and a second surface. For film samples, the surfaces are considered to be at least locally parallel. That is that the areas of the first and second sample surfaces that encounter the radiation beam are substantially parallel, even if the surfaces as a whole are not entirely parallel.

This condition of parallel surfaces is accommodated for liquid samples by loading the liquid sample into a cuvette with substantially parallel walls, discussed in more detail below.

The detection device may be a photo-detector which comprises an array of radiation sensors. The accuracy of the measurement of **n** and **L** is dependent on the resolution of the detection device. Charge coupled device (CCD) cameras offer high resolution and sensitivity for such applications. The CCD camera can comprise either a single two-dimensional sensor or an array of sensors. A CCD camera and appropriate software can be used to locate the beam spots before and after deflection or reflection. The displacement 28 can be deduced from the center of gravity of the beam spot.

The detection device is preferably positioned so that reflected or transmitted beams are completely intercepted. A minimum lateral distance from the source and the detection device may be calculated for this purpose. The variation in the lateral distance of the transmitted and reflected beams is a function of the incidence angle. Beam divergence values may also be considered when positioning the detection device.

The detection device may be connected to a computer or other data handling device. The computer may be programmed to measure the distances d_1, d_2, d_x or d_y from the detection device and may further be used to calculate **L** and **n** from the appropriate set of equations.

Now referring to FIG. 4, an additional embodiment of the present invention is illustrated wherein the sample is contained in a transparent cuvette. This method employs the same imaging technique as discussed above using reflected beams. This method allows the measurement of other physical constants of fluid samples.

A radiation source 12 transmits a beam 13 at an incident angle 16, and a portion of the beam 1 is reflected of the first cuvette wall 42'. Another portion of the beam 2 is reflected off the cuvette wall 42' to sample 40 interface. Another portion 3 of the same beam is reflected off the interface between the sample 40 and the second cuvette wall 42, and another reflected beam 4 results from a reflection off the cuvette wall 42 to air interface. The total-internal reflection angle on either the cuvette-sample or sample-cuvette interface is avoided when the incident light angle is lower than the critical angle for either interface.

The geometry of the different reflections captured in a detector plane 46, and imaged on a display 48, illustrate how the principle applied to the film samples can be extended to calculate the thickness of the cuvette walls through R' measurement. The measurement of R and R' allows the estimation of the thickness' of the sample cuvette walls and the fluid within in a similar manner as applied above. This extension of Snell's Law includes measuring the relative intensities of beams 1 to 3 to estimate the absorption coefficients of the cell and of the fluid. Further coefficients, such as the reflection and transmission coefficients of each interface can also be estimated using two or more incident beams of various intensities. An intensity-modulated laser beam may be advantageously employed for this purpose.

For optimal performance in measuring **L** and **n**, a low-divergence laser beam is preferred. Low-divergence for these purposes is on the order of mrads in magnitude. If higher divergence beams are to be used, the beam sizes should preferably be smaller than the detection matrix dimensions. For CCD camera detectors, the detection matrix dimensions are typically 10x10 mm^2, however 30x30 mm^2 are currently available in the market. The orientation of the source, sample and detector is preferably optimized in

both the transmission and reflection modes to minimize any decrease in detector resolution caused by beam divergence.

The measurement method of the present invention is illustrated by the following examples but is not intended to be limited thereby:

EXAMPLES

An exemplary measurement was preformed with a HeNe laser at 544 nm as the radiation source, and a sample placed on a platform with angular motion capability about the horizontal. The platform was on a rotation stage with free motion of 360° and rotational resolution of 1°. The beam displacement was monitored by a CCD camera with pixel separation of approximately 13 μm and each image was downloaded to a computer. The laser beam was attenuated with two polarizers from its original 1.5 mW to less than 1 μW so as not to saturate the CCD camera.

The displacement d was measured by subtracting the horizontal coordinate of the beam spot on the CCD at $\alpha=0°$ from the similar coordinate at another α value. Two values of d are measured at two different angles for each sample.

The system of two equations derived from EQN. 1 with the two sets (α_1, d_1) and (α_2, d_2) was solved numerically, leading to a unique solution for **L** and **n**.

Several samples with different thickness' and refractive indices were analyzed according to this method and the results appear in the following tables for **L** (Table 1) and n (Table 2).

$L_{measured}$(mm)	0.24	0.99	1.03	3.06	3.07	5.84	10.02
Precision(%)	3.7	1.1	2.6	1.9	2.4	2.7	1.0

Table 1: The measurements and corresponding errors of the thickness **L** of each sample.

$N_{measured}$	1.48	1.51	1.58	1.60	1.60	1.61	1.74
Precision(%)	1.3	3.2	6.8	4.1	6.4	4.7	17.0

Table 2: The errors on the measurements of the refractive index n of each sample.

The errors for the thickness estimations are due to a systematic error included by the imaging software. The software used for these examples includes a bias displacement to each spot location when snapping a single image frame. As a result, a relatively high error on the estimation of the displacements was introduced. These errors appear to be more significant in the estimation of the refractive index as the numerical program employed determines **n** from the estimated value for **L**. The estimation error is amplified accordingly in the refractive index measurement. Therefore, lower errors on the estimations are expected with software refinement.

Furthermore, a spatial filter may be used in the laser beam path before the sample to generate a smooth gaussian laser beam cross section that may be fitted correctly with a software to allow precise localization of the beam spot on the CCD camera.

Those having the benefit of the above description of my invention may provide numerous such modifications of the invention. These modifications are to be construed as being encompassed within the scope of the present invention as set forth in the appended claims.